Electropolish Experiment 60303- Sulfur Precipitation

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Introduction DRAFT!!!!!!

An electropolish experiment was performed in the off-line chemistry room to determine if sulfur could be precipitated and collected onto a witness sample for analysis. Goal of this experiment was to determine if sulfur particles collected on the witness sample would cause field emission and to determine if the experimental setup was adequate to perform additional experiments to determine under what voltage and current parameters sulfur would be precipitated.

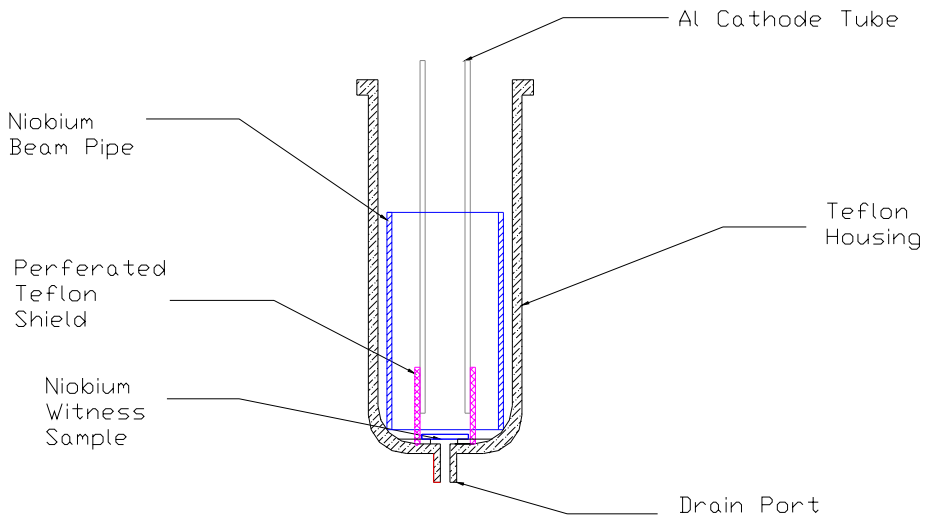
Assumptions:

1. Over voltage condition would present the highest probability for sulfur to be produced with oxygen bubbles forming at the anode.
2. To identify if the apparatus was correct for this experiment and to allow for analysis for field emission, a niobium witness sample would be used to collect the sulfur but would not play an active role in the electropolish itself.
3. In order for the sulfur to collect into clusters the process would be cooled to first and allowed some time to form.
4. The volume of sulfuric acid and the anode surface area should be as large as possible with in the setup to be successful on the first try
5. in order to collect sulfur on the witness sample the entire volume of electrolyte would be drained past the sample in increase the likely hood of the sulfur clusters residing on the witness sample

Experimental Procedure

1. Using existing hardware, a Teflon ® filter housing was mounted into a stainless steel frame to hold it upright and in place. This frame was then placed into the fume hood
2. Two witness samples S1 and S2 were then electropolished to improve their surface finish for the experiment. S1- would be used as a control and S2 – which had 0.1751g of niobium removed as the witness sample. Surface area ~ 28cm^2
3. S2 was placed in the base of the housing above the drain port.
4. A niobium beam pipe section was inserted into the housing and the anode contact was connected to it (positive contact). The surface area of the tube was ~ 318cm2 inside and the same outside.
5. An aluminum tube approximately 9 inches was wrapped at the base with perforated Teflon sheet to prevent contact with the Niobium witness sample at the base of the housing
6. A standard 1.3 inch diameter aluminum tube, was inserted in the center of the niobium tube for the cathode and connected to the negative contact.
7. Electrolyte was mixed for one gallon of solution with the standard mixture of 10% HF (49%) and 90% H2SO4 (98%) by volume.

8. An isolation valve was added to the drain line to allow filling of the housing with electrolyte.
9. The housing was filled with electrolyte to the top of the niobium tube
10. Power supply was setup to constant voltage and adjusted for a current of 18-20 amps for one hour.
11. Power was shut off and housing was water cooled for a few minutes
12. The housing was drained of all electrolyte
13. The housing was then filled with fresh electrolyte and power turned back on for 10 more minutes
14. The housing was then cooled for a few minutes with DI water and then drained past the witness sample again.
15. Cathode was removed
16. Niobium beampipe was removed
17. Housing with witness sample was rinsed out with DI water
18. Housing was laid flat and witness sample slid out on to a gloved hand and blown dry with nitrogen gas and inserted into a clean plastic sample container.



Electropolish Experimental Setup
(Sulfur Production)

Observations:

1. A dark film formed on the acid surface which ended up on the inside of the housing from the electrolyte level to the bottom when draining. Question is whether the film is on the surface of the witness sample and if so what it is.

2. The film was not removed by flushing with DI water or micro and DI water.
3. Isopropyl alcohol on a cleanroom wipe was used remove the film from the housing by wiping.
4. The housing smelled of sulfur before cleaning and after Micro and DI water rinsing.
5. The Copper anode contact was etched during the experiment when the electrolyte gas bubbles rose above the contact. Question is this the source of the film color and the reason it formed
6. Chemroom environment conditions are not clean and may add contamination to the sample during handling of the sample.
7. Water quality of the DI water unknown.

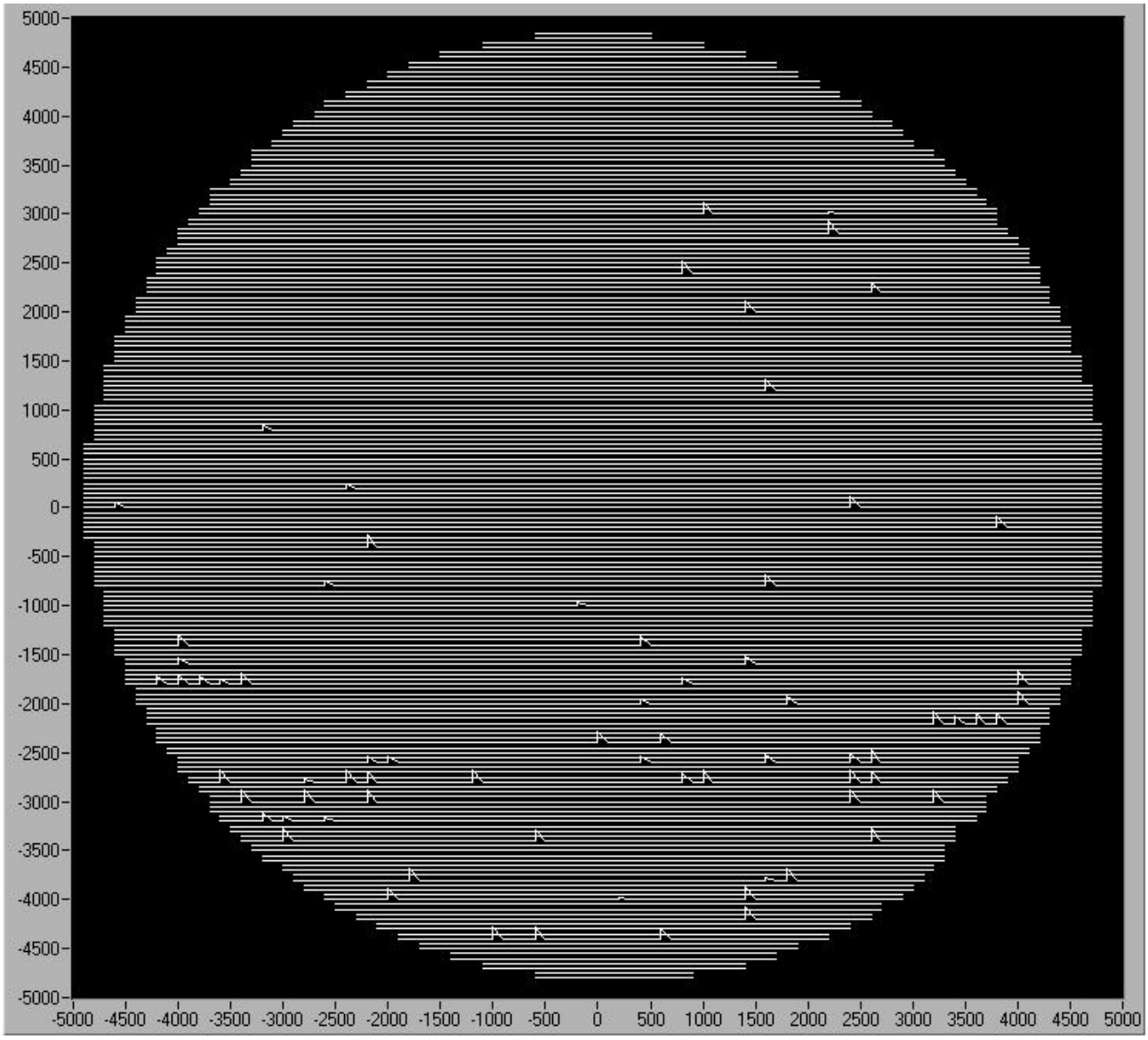
Experimental Results:

1. Sulfur clusters were found on the niobium witness samples
2. Witness Sample had many field emission sites including , Cu, Si, S, Al, Ti, Cl, Na
3. Sulfur cluster was one of the last elements to field emit at ~ 120MV/m and the lowest was ~86 MV/m

SEM DATA: see below

Follow-up Experiments:

- **It would be nice to repeat the experiment for several points on the IV curve and test for sulfur formation to see when does it start**
- **Additional question are:**
  - **How does the volume of H2SO4 and HF effect this production of Sulfur?**
  - **Is there a film left on the cavity surface after processing?**
  - **How does the distance of the cathode affect this process?**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